



UNIVERSITI PUTRA MALAYSIA

**HOPANES AND POLYAROMATIC HYDROCARBONS FROM 21 2T
OIL AS MOLECULAR MARKERS FOR DETECTING THE SOURCES
OF HYDROCARBON POLLUTION**

WONG YOON LEE.

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HYDROCARBON POLLUTION**

By
WONG YOON LEE

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfillment of the Requirements for the Degree of Master of Science**

July 2005



TO MY DEAREST FAMILY AND FRIENDS

Abstract of thesis presented to the Senate of University Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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MOLECULAR MARKERS FOR DETECTING THE SOURCES OF
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WONG YOON LEE

July 2005

Chairman: Associate Professor Mohamad Pauzi bin Zakaria, PhD

Faculty: Environmental Studies

Rapid growth of population, urbanization and industrialization in Malaysia led to sharp increase in numbers of motor vehicles. Therefore motor oils are potential land-based sources that contributed to hydrocarbon pollution in Malaysia. Two-cycle lubricating oil (2T oil) is a lubricating base oil specially used in two-stroke motorcycle engines. Lubricating base oils are complex mixtures of hydrocarbons. This study focused on alkanes, hopanes and PAHs in 2T oil due to their importance as molecular markers and their combination would be a powerful tool to distinguish input, sources and transport pathway of hydrocarbon pollution. The objective of this study is to analyze alkanes, hopanes and PAHs from 2T oil. Alkanes, hopanes and PAHs from 2T oil were used as molecular markers and were applied for detecting the sources of hydrocarbon pollution and the transport pathway of the 2T oil.

Three kinds of commonly use fresh 2T oil samples were purchased from local market. Used 2T oil sample was collected with pre-cleaned cotton buds from two-

stroke motorcycles tail pipes at Central Parking of Universiti Putra Malaysia. The cotton buds were rinsed with 3:1 Hex/DCM. The extract was fractionated and analyzed for PAHs, Hopanes and alkanes by gas chromatography mass spectrometry (GC-MS). The result indicates that used 2T oil contained much higher concentration of PAHs than fresh 2T oil due to formation of PAHs in concentration during high temperature combustion in the engine (Rafael, 1989). Application of the source identifiers, UCM, Pr/Ph, CPI, C_{29}/C_{30} , $\Sigma C_{31}-C_{35}/C_{30}$, MP/P and H/L-PAH suggested that hydrocarbon from fresh and used 2T oil both were contributing to the street dust and air particulate in some level of contamination through the exhaust of two-stroke motorcycles. Combination of the alkanes, hopanes and PAHs fingerprints comparison among fresh and used 2T oil, river, inshore, offshore, street dust and air particulate showed that both hydrocarbons from fresh and used 2T oil were significant in coastal environment of Malaysia and they had been potentially transported via street dust and air particulate. PAHs from 2T oil might be discharged to river and inshore coastal zone of Malaysia through street dust and air particulate via lateral and vertical transportation.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**HOPANE DAN POLIAROMATIK HIDROKARBON DARIPADA MINYAK
2T SEBAGAI PENANDA-PENANDA MOLEKUL UNTUK PENGESANAN
SUMBER PENCEMARAN HIDROKARBON**

Oleh

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Perkembangan populasi, pembandaran dan pengindustrian yang pantas di Malaysia telah menyebabkan peningkatan dalam jumlah kenderaan yang pantas. Maka, minyak engin merupakan sumber daratan yang berpotensi menyumbangkan kepada pencemaran hidrokarbon di Malaysia. Minyak pelicin dua lejang (minyak 2T) ialah sejenis pelincir yang digunakan khasnya dalam enjin motosikal dua lejang. Minyak pelicin adalah campuran hidrokarbon yang rumit. Kajian ini memberi tumpuan atas alkane, hopane dan Polisiklik Aromatik Hidrokarbon (PAHs) dalam minyak 2T disebabkan mereka adalah penanda molekul yang penting dan kombinasi mereka akan menjadi sesuatu alat yang hebat untuk membezakan kemasukan, sumber dan perjalanan pencemaran hidrokarbon. Objektif kajian ini adalah untuk menganalisis alkane, hopane dan PAHs daripada minyak 2T. Alkane, hopane dan PAHs adalah digunakan sebagai penanda molekul untuk menentukan sumber pencemaran hidrokarbon.

Sebanyak tiga jenis sample minyak 2T mentah yang biasa diguna dibeli dari pasaran tempatan. Sample minyak 2T terpakai dikumpul dari paip belakang motosikal dua lejang dengan mengguna kapas yang bersih di tempat letak kereta utama Universiti Putra Malaysia (UPM). Kapas-kapas tersebut dibilas dengan 3:1 Hex/DCM. Ekstrak daripada bilasan tersebut dipisahkan dan dianalisis untuk PAHs, Hopane dan alkane dengan kromatografi gas spektrometri jisim (GC-MS). Keputusan kajian ini menunjukkan minyak 2T terpakai mengandungi kepekatan PAHs terlampau lebih jika berbanding dengan minyak 2T mentah. Ini disebabkan pembentukan PAHs dalam kepekatan semasa pembakaran pada suhu yang tinggi dalam enjin (Rafael 1989). Pemakaian penentu-sumber (UCM, Pr/Ph, CPI, C_{29}/C_{30} , $\Sigma C_{31}-C_{35}/C_{30}$, MP/P and H/L-PAH) jelas mencadangkan minyak 2T mentah dan terpakai telah menyumbangkan kepada abuk jalan dan zarah udara melalui eksos motosikal dua lejang pada garisan pencemaran yang tertentu. Kesemua perbandingan penanda alkanes, hopanes dan PAHs di antara minyak 2T mentah, minyak 2T terpakai, endapan sungai, pantai dalam dan luar Selat Melaka, abuk jalan dan zarah udara menunjukkan minyak 2T mentah dan terpakai berpotensi diangkut ke sediment Malaysia melalui abuk jalan dan zarah udara. Sumber PAHs dari minyak 2T kemungkinan discas ke dalam endapan sungai dan persekitaran pantai melalui perjalanan secara sisi dan menegak.

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I certify that an Examination Committee met on 20th July 2005 to conduct the final examination of Wong Yoon Lee on her Master of Science thesis entitled “Hopanes and Polyaromatic Hydrocarbons from 2T Oil as Molecular Markers for Detecting the Sources of Hydrocarbon Pollution” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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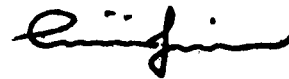
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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently for any other degree at UPM or other institutions.



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Date: 17/10/2005

TABLE OF CONTENTS

	Page
DEDICATION	ii
ABSTRACT	iii
ABSTRAK	v
ACKNOWLEDGEMENTS	vii
APPROVAL	viii
DECLARATION	x
LIST OF TABLES	xiii
LIST OF FIGURES	xiv
LIST OF ABBREVIATION	xviii
 CHAPTER	
1 INTRODUCTION	1
1.1 General introduction to oil pollution	1
1.2 The significance of this study	12
1.3 The objectives of this study	19
 2 LITERATURE REVIEW	20
2.1 Lubricating base oil	20
2.2 Two-stroke engine and 2T oil	23
2.3 Molecular markers	25
2.4.1 Alkanes	27
2.4.2 Hopanes	30
2.4.2 Polycyclic Aromatic Hydrocarbons (PAHs)	31
2.4 Transport pathway of hydrocarbon	33
2.6 Sources and transport pathway of PAHs	35
2.7 Behavior of PAH in atmosphere	39
 3 METHODOLOGY	43
3.1 Chemicals and Material	43
3.1.1 External Standard Mixture for alkanes	43
3.1.2 Internal Injection Standard and Standard Mixture for Hopanes	43
3.1.3 Surrogate Internal Standard (SIS), Internal Injection Standard (IIS) and native standard mixture for PAHs	44
3.1.4 Organic Solvents	48
3.1.5 Glass Equipment	48
3.1.6 1 st and 2 nd step Silica Gel	49
3.1.7 Sodium sulphate (Na ₂ SO ₄) anhydrous	50



3.1.8	Activated Copper (Cu)	50
3.1.9	Pre-cleaned Cotton Buds	51
3.2	Sampling Site	51
3.3	Sample collection	54
3.3.1	Fresh 2T oil (source material)	54
3.3.2	Used 2T oil (source material)	54
3.4	Analytical Procedure	56
3.4.1	Fresh and Used 2T oil	56
3.4.2	Street Dust and Air Samples	61
3.5	Instrumental Analysis	61
3.5.1	Instrumental Analysis of n-alkanes	62
3.5.2	Instrumental Analysis of Hopanes	62
3.5.3	Instrumental Analysis of PAHs	63
3.6	Calculation of concentration in the sample	64
3.6.1	Alkanes concentration	64
3.6.2	Hopanes concentration	65
3.6.3	PAHs concentration	70
4	RESULTS AND DISCUSSION	76
4.1	Alkanes composition in fresh and used 2T oil, air particulate and street dust	76
4.2	Source Identification using UCM, Pr/Ph ratio and CPI	79
4.3	Hopanes composition in fresh and used 2T oil, air particulate and street dust	83
4.4	Sources Identification by using source identifiers, C_{29}/C_{30} ratio and $\Sigma C_{31}-C_{35}/C_{30}$ ratios	84
4.5	The PAH content in fresh and used 2T oil	88
4.6	Sources Identification: Application of MP/P ratio and H/L-PAH ratio	93
4.7	Contribution of fresh and used 2T oil to street dust and air particulate	97
4.8	Understanding the transport pathway of fresh and used 2T oil from land to ocean	99
5	CONCLUSION AND RECOMMENDATION	115
5.1	Conclusion	115
5.2	Recommendation	117
	REFERENCES	119
	APPENDIX	129
	BIODATA OF THE AUTHOR	162

LIST OF TABLES

Table	Page
1.1 Oil Spills Occurring in the World	3
1.2 Oil Spills Accidents Occurring in Strait of Malacca, Malaysia	3
1.3 Some example structure of n-alkanes	15
1.4 15 EPA priority PAH pollutants	18
3.1 Name, types, molecular structure of Hopanes Internal Injection Standard (IIS) and compound (C) in the hopanes standard mixture	45
3.2 The PAH Compounds, Internal Injection Standard (IIS) and Surrogate Internal Standard (SIS) inside the PAH Standard Mixture	47
3.3 Target alkanes compounds analyzed in this study and corresponding standards for respond factor of the compounds.	66
3.4 Name, abbreviation and molecular structure of hopane compounds analyzed in this study.	68
3.5 The corresponding deuterated surrogates used to quantify each target PAH compounds recovery rates	72
4.1 Hopanes, PAHs and Alkanes Composition for Fresh and Used 2T oil, Air Particulate, Street Dust, Crude Oil (MECO; SEACO), Asphalt, Fresh and Used Crankcase Oil, and Malaysia Sediment (River; Inshore; offshore),	77



LIST OF FIGURES

Figure		Page
1.1	The percentage of the main categories of sources contribute to the total worldwide annual released of petroleum about 1.3 million tones per year (NRC, 2002)	5
1.2	Contribution of different sources to oil pollution in the South China Sea (GESAMP, 1993)	5
1.3	Example of Hopanes/Pentacyclic Triterpanes molecular structure	17
3.1	(a) World map showing location of Malaysia (b) Area enlarged map of Peninsular Malaysia, showing location of Straits of Malacca, Kuala Lumpur and Universiti Putra Malaysia (UPM).	52
3.2	(a) Universiti Putra Malaysia (UPM) at Selangor State of Malaysia. (b) Area enlarged of UPM's main campus, about 23km to the south of Kuala Lumpur; (c) Sampling location for used 2T oil sample at Central Parking of UPM.	53
3.3	(a) Fresh 2T oil Sample, Shell Advance SX 2T; and (b) Photography of the used 2T oil sample collection	55
3.4	Analytical procedure for fresh and used 2T oil in this study	57
3.5	Molecular Structure and name of 15 target PAH compounds analyzed in this study.	71
3.6	Schematic chromatograms and peaks in the standard mixture solution (a) and sample solution (b).	73
4.1	Total Ion Current chromatogram of Alkane for, (a) Fresh 2T oil, Castrol; (b) Used 2T oil; (c) Street Dust, SD; (d) Air Particulate, Sample #4.	80
4.2	mz 191 mass chromatogram of hopanes in: (a) Fresh 2T oil, Castrol; (b) Used 2T oil; (c) Street Dust; (d) Air Particulate, Sample#2.	85
4.3	Hopane Gas Chromatograms of SEACO and MECO	86



4.4	Total PAHs of fresh and used 2T oil	89
4.5	PAH profile of (a) Used 2T oil, and (b) Fresh 2T oil (Petronas), showing that the composition of 2-3 rings PAHs	92
4.6	The Specific formula of MP/P ratio used in this study	94
4.7	PAH Profile of (a) fresh 2T oil; (b) Used 2T oil; (c) Air Particulate (Sample #4) and (d) Street Dust (SD).	96
4.8	(a) Pr/Ph Comparison Diagram; and (b) CPI Comparison diagram. Sample fresh 2T (Shell, Castrol and Petronas); used 2T oil; Klang Estuary river sediment, (St G, St. C); street dust (KL-1, KL-2, KL-3) from Zakaria <i>et al.</i> 2000; Street dust (SD) and air particulate (#1, #2, #3 and #4).	101
4.9	Hopanes composition in fresh and used 2T oil; sediment samples of Klang Estuary, Klang Coast, and the Straits of Malacca, street dust samples (KL-1, KL-2 and KL-3) from Zakaria <i>et al.</i> (2000); street dust, SD and air particulate samples (a) C_{29}/C_{30} ratio; and (b) $\Sigma C_{31}-C_{35}/C_{30}$ ratio	102
4.10	C_{29}/C_{30} vs. $\Sigma C_{31}-C_{35}/C_{30}$ ratio diagram	105
4.11	MP/P ratio Comparison Diagram shows MP/P ratio of the sample analyzed in this study (fresh and used 2T oil) compared with samples in Zakaria <i>et al.</i> , 2002 (river sediments; inshore sediment; offshore sediment; street dust, KL-1, KL-2, KL-3); Suzaivanti, 2004 (street dust, SD); and Chong, 2004 (Air particulate).	107
4.12	H/L-PAH Comparison Diagram shows H/L-PAH for samples analyzed in this study (fresh and used 2T oil) compared with samples in Zakaria <i>et al.</i> , 2002 (river sediments; inshore sediment; offshore sediment; street dust, KL-1, KL-2, KL-3); Suzaivanti, 2004 (street dust, SD); and Chong, 2004 (Air particulate).	109
4.13	PAHs Comparison Profiles: (a) used 2T oil; (b) fresh 2T oil, Castrol; (c) river sediment (Klang Estuary), St. A; (d) inshore sediment (Klang Coast), St 2K; (e) offshore sediment (The Straits of Malacca), St. 8; (f) street dust, SD; (g) air particulate, sample #4. Data for (c) to (e) was kindly provided by Zakaria <i>et al.</i> (2002), data for (f) was provided by Chong (2004) and data (g) was provided by Suzaivanti (2004).	110
4.14	The proposed model of the transport pathway of used 2T oil to the terrestrial and ocean environment.	112

Appendix A

A1	Laboratory Analysis of fresh 2T oil	130
A2	Used 2T oil Collection	130

Appendix B

B1	1 st Step Column Chromatography	131
B2	2 nd Step Column Chromatography	131
B3	Rotary Vacuum Evaporator	132
B4	Gas Chromatography/Mass Spectrometry (GC/MS)	132

Appendix C

C1	Total Ion Current Trace and Peak Reports of Alkane External Standard Mixture	133
C2	Total Ion Current Trace and Peak Report of Alkane in Fresh 2T oil (Castrol)	134
C3	Total Ion Current Trace and Peak Report of Alkane in Used 2T oil	135
C4	Total Ion Current Trace and Peak Report of Alkane in Street Dust (Sample SD)	136
C5	Total Ion Current Trace and Peak Report of Alkane in Air Sample (Sample#4)	137
C6	m/z 191 Mass Chromatogram and Peak Report of Hopane + Alkane Standard Mixture for Fresh 2T Oil	138
C7	m/z 191 Mass Chromatograms and Peak Report of Hopane in Fresh 2T oil (Castrol)	139
C8	m/z 191 Mass Chromatogram and Peak Report of 2ppm Hopane Standard Mixture with IIS for Used 2T Oil.	140
C9	m/z 191 Mass Chromatograms and Peak Report of Hopane in	141



Used 2T Oil with IIS

C10	m/z 191 Mass Chromatogram and Peak Report of Hopane + Olenanes Mixture, (4ppm), for Street Dust and Air Particulate Samples.	142
C11	m/z 191 Mass Chromatograms and Peak Report of Hopane in Street Dust (Sample SD) with IIS	143
C12	m/z 191 Mass Chromatograms and Peak Report of Hopane in Air Particulate Sample (Sample#2) with IIS	144
C13	GCMS Chromatogram and Peak Report of PAH Standard Mixture with IIS and SIS	145
C14	GCMS Chromatogram and Peak Report of PAH in Fresh 2T oil (Petronas) with IIS and SIS	146
C15	GCMS Chromatogram and Peak Report of PAH in Used 2T Oil with IIS and SIS	147

Appendix E

E1	NEWSPAPER CUTTING: NEW SUNDAY TIMES, NOVEMBER 14, 2004.	160
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LIST OF ABBREVIATION/GLOSSARY OF TERMS

An	Anthracene
Acenaphth-d ₁₀	Acenaphthene-deuterated-10
BaA	Benzo[a]Anthracene
BaPy	Benzo[a] pyrene
BeAceph	Benzo[e] acephenantherene
BePy	Benzo[e]Pyrene
BkF	Benzo[k]Fluoranthene
Chry	Chrysene
Chry-d ₁₂	Chrysene-deuterated-12
C ₂₉ /C ₃₀	Ratio of 17 α ,21 β (H)-30-norhopane to 17 α ,21 β (H)-30-hopane
Σ C ₃₁ -C ₃₅ /C ₃₀	Ratio of sum of C31 homohopane to C35 homohopane relative to 17 α ,21 β (H)-30-hopane
CPI	Carbon Preference Index
Cu	Copper
DBahA	Dibenzo[a,h] anthracene
DBT	Dibenzothiophene
DCM	Dichloromethane
Fluo	Fluoranthene
GC/MS	Gas Chromatography Mass Spectrometry
H/L-PAH	High Molecular Weigh PAH/Low Molecular Weight PAH
Hex	Hexane
HMW	High Molecular Weight
IIS	Internal Injection Standard
LABs	Linear Alkyl Benzenes
LMW	Low Molecular Weight
L/H-alkane	Low Molecular Weight Alkane/High Molecular Weight Alkane
MAAn-2	2-methylAnthracene
MeOH	Methanol
MECO	Middle East Crude Oil



MP-1	1-methylphenanthrene
MP-2	2-methylphenanthrene
MP-3	3-methylphenanthrene
MP-9	9-methylphenanthrene
MP/P	Methylphenanthrene/Phenanthrene
MPy-1	1-methylPyrene
Napth-d ₈	Napthalene-deuterated-8
Na ₂ SO ₄	Sodium Sulphate Anhydrous
PAHs	Polycyclic Aromatic Hydrocarbons
Pery-d ₁₂	Perylene-deuterated-12
Phe	Phenanthrene
Phe-d ₁₀	Phenanthrene-deuterated-10
Pri	Pristane
Pr/Ph	Pristane/Phytane
Py	Pyrene
SEACO	South East Asian Crude Oil
SIS	Surrogate Internal Standard
T _m /T _s	17 α -22, 29,30-trisnorhopane / 18 α -22, 29,30-trisnorhopane
UCM	Unresolved Complex Mixture
UPM	Universiti Putra Malaysia
1,4-DCB-d ₄	1,4-dichlorobenzene-deuterated-4
2T Oil	Two cycle Lubricating Oil

CHAPTER 1

INTRODUCTION

1.1 General introduction to hydrocarbon pollution

Nowadays, petroleum is a term used as common denotation for crude oil (mineral oil) and natural gas. Then petroleum also is a collective term for hydrocarbon whether solid, liquid or gaseous. Crude oil is a complex mixture of thousands of different chemical components, mainly organic compounds which usually made up about 95% of the crude oil. Before used as fuel or as raw material in the petrochemical industry, crude oil is refined into different fractions. At the refinery, crude oil is separated into light and heavy fractions such as natural gas, raw gasoline, intermediate distillates, heavy distillates and residues, which are then converted into various product, such as petrol, fuels, diesel oil, lubricating oil, waxes and asphalt.

Oil, refined product and pyrogenic hydrocarbons are the most frequently discovered contaminants in the environment (Wang and Fingas, 2003). When crude oil or refined petroleum product are accidentally released to the environment, it will cause oil or hydrocarbon pollution in the environment. When petroleum hydrocarbons introduce to an ecosystem, they alter most of the ecological process and result in long-term chronic effects on marine organisms (Elizabeth, NST, 30 May 2004)).

Sources of oil input to the marine environment are often divided into natural, land-based and sea-based. Natural sources are sources come from natural seeps. Land-based sources are sources discharges of untreated or insufficiently treated of oil from coastal refineries, oil terminal, etc. While sea-based sources are sources comes from accidental oil spills, oil platforms (blowouts), pipelines, operational discharges of oil, dumping of oily waste, ship-related activities (i.e. motor boat with two-stroke engine), emission of gaseous hydrocarbons from tankers and pleasure craft, etc.

Accidental, operational discharge and spills of oil from ships, especially tankers, offshore platforms and pipelines, is the most obvious and visible cause of oil pollution of the marine environment. Table 1.1 shows the current oil spills accidents occurring in the world. Table 1.2 shows the current oil spills accidents occurring in the Straits of Malacca, Malaysia.

However, natural sources (large quantities) and land-based sources account for a large part of the total annual input of oil to the marine environment. An estimated 21 million barrels of oil run into the oceans each year from street run-off, effluent from factories and from ships flushing their tanks (Elizabeth, NST, May 30, 2004).

According to the recent report published in 2002 by National Research Council (NRC) of the U.S. National Academy of Sciences, the total average worldwide annual input of oils to the marine environment is about 1.3 million tones per year.

Table 1.1: Oil Spills Occurring in the World

Year	Name	Accident Place	Cause	Amount (Millions Gallons)
1967	Torrey Canyon	English Channel	Grounding	38.2
1972	Sea Star	Gulf of Oman	Collision	37.9
1977	Hawaiian Patriot	Pacific Ocean	Fire	31.2
1978	Amoco Cadiz	Brittany Coast, France	Grounding	68.7
1979	Ixtoc-1 Oil Well	Gulf of Mexico	Blowout	140
1979	Atlantic Empress	Trinidad and Tobago	Collision	42.7
1980	Well	Southeast of Tripoli	Operations	42.0
1980	Irenes Serenade	Greece	Grounding	36.6
1981	Storage Tanks	Shuaybah, Kuwait	Operation	31.2
1983	Castillo de Bellver	64km off Table Bay, South Africa	Fire	78.5
1989	Exxon Valdez	Prince William Sound, Alaska, USA	Human Error	11.0
1991	Terminals, Tankers, Pipelines, Persian Gulf	Kuwait & Arabian Gulf	Gulf War	240
1992	Oil Well Fergana Valley	Uzbekistan	Operations	88.0
1993	Braer	Shetland Island	Grounding	25.0
1994	Pipeline Kharyaga- Usinsk	Russia	Burst Pipe	30.7
1996	Sea Empress	Wales	Grounding	24.0
1999	Maltese Tanker Erika	South of Brest, France	Fierce Storm	2.8

Source: Oil Spill History (marinegroup.com), 2004

Table 1.2: Oil Spills Accidents Occurring in Strait of Malacca, Malaysia

Year	Name of Ships	Cause	Amount (tons)
1975	SHOW MARU	Grounding	400
1976	DEIGO SILANG	Collision	4000
1981	MT OCEAN TRESURE	Human Error	60
1986	BRIGHT DUKE	Collision	NA*
1993	NAGASAKI SPIRIT	Collision	13300
1997	ANTAI	Grounding	235

Source: Razif Ahmad (1995)

*NA – Not Available

The major categories of sources that contributed to the input are shown in Figure 1.1. It shows that natural seeps and discharge from consumption of oil (land-based source) contribute larger input (up to 80 %) of oil pollution to the worldwide seas. While, Figure 1.2 shows the relative contribution of different sources to the oil pollution in the South China Sea (GESAMP, 1993). 50% of the oil pollution contribution in South China Sea comes from land-based sources (municipal and industrial sources). Furthermore, it has been estimated that on global scale, up to 70% of pollution in the seas originated from land-based sources (UNEP, 1990).

Malaysia is geographically made up of two regions, West Malaysia (peninsular Malaysia) and East Malaysia (Sabah and Sarawak), which are separated by South China Sea. It occupies a total land area of approximately 332, 800 square km and total sea area of approximately 598,540 km (The World Factbook, 2004). Being located between longitude 1°- 7° North and latitude 100° - 120° East, Malaysia is influenced by the equatorial environment and is well outside volcanic, tornado and severe drought belts. Strategically, the country is centrally located to various international air and sea transport and communication routes. The strategic location as a major international shipping lane and the concentration of agriculture, industry and urbanization were predominate on the west coast of Peninsular Malaysia making the Straits of Malacca a great variety of environmental stresses.

Malaysia is having a rapid social-economic growth in the last two decades to achieve a developed country by the year 2020. Therefore, urbanization, industrialization and population are increasing rapidly especially in the capital city

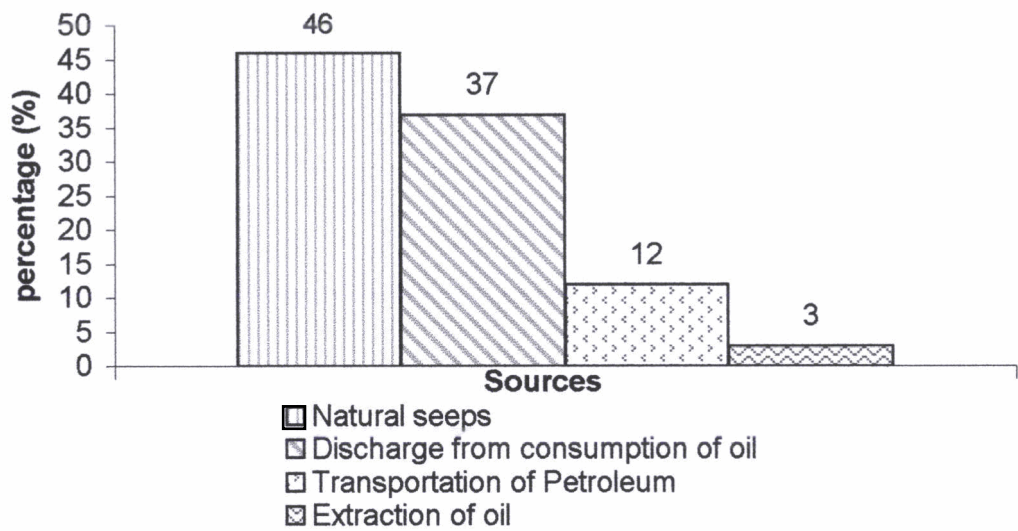


Figure 1.1 : The percentage of the main categories of sources contribute to the total worldwide annual released of petroleum about 1.3 million tones per year (NRC, 2002).

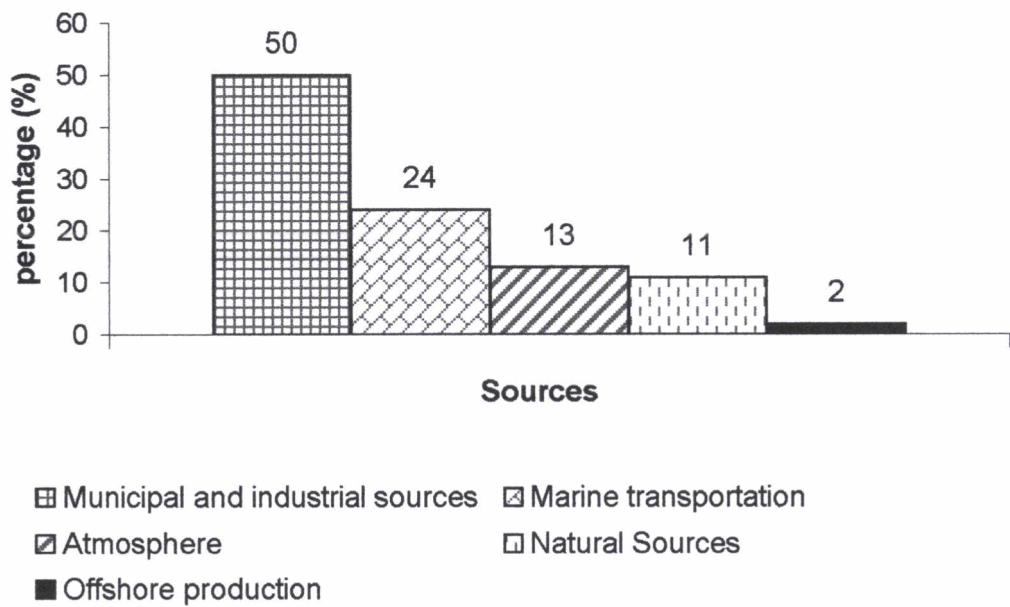


Figure 1.2: Contribution of different sources to oil pollution in the South China Sea (GESAMP, 1993)